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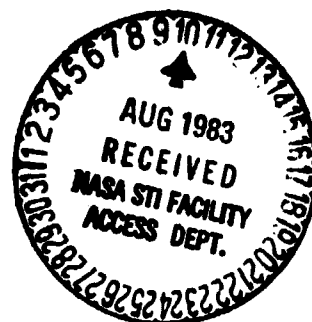
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THE PREPARATION OF STEATITE SUSPENSION FOR SPRAY DRYING

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rozprasovaci suseni," Sklar a keramik, Vol. 32,
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16. Abstract Liquifying agents were investigated for preparation of highly concentrated steatite suspensions which are to be spray-dried. Organic additives for improving the molding properties and strength of green compacts are described. Demands on properties of the spray-dried granules are defined with regard to shrinkage of the molded compacts.					
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PREPARATION OF STEATITE SUSPENSION FOR SPRAY DRYING

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The article presents a summary of findings regarding the effects of liquefying agents in preparation of a highly concentrated steatite suspension for spray drying. It describes the use of organic substances with bonding, plastification and separation properties for improving the compressibility and strength of green compacts. It formulates the requirements on properties of granulates prepared by spray drying with a view to shrinkage of compacts from devices designed for making compacts from granules prepared by premolding.

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Introduction

Steatite products were originally molded from granules prepared by crushing tablets obtained by molding materials from dried filter press cakes. The molding mixture obtained by screen sorting had a bulk density weight of 1,600 to 1,800 g/l. Molding devices were designed to provide shrinkage of corresponding values.

The original technology became somewhat changed by use of spray drying. That eliminated filter pressing, premolding and preparation of granules from premolded tablets. Part of the instrumentation and equipment--drum mills, traps, molding devices--remained unchanged. Nevertheless, this development brought about a number of technical problems. One requirement was continued use of the unchanged molding devices. This called for moldings prepared from spray dried granulate to have the same shrinkage, identical or better mechanical strength, surface appearance, etc., as moldings prepared from premolded granulate. As the conventionally prepared molding mixture had a high degree of bulk density, it was also up to spray drying to come up with a granulate of comparable bulk density which can be done from a suspension containing a large share of dry matter.

*Numbers in the margin indicate pagination in the foreign text.

The objective was to propose a process for preparation of stearite suspensions of a high concentration and good fluidity suitable for spray drying.

A brief report on grinding of concentrated suspensions has already been published [1].

The principle of liquifying consists in controlling the process of coagulation and dispersion. Producing a suspension of good fluidity calls for generation of an adequate repelling force between particles. The source of this force is the electrical charge of the particles which attracts in the dispersion medium ions with the opposite charge. This creates an equilibrium charge on the surface of the particles. This system in a solvation medium is as a whole electroneutral and the whole unit, i.e., the solid particle and its ion shell is referred to as a micelle. Of course, the ion shell of a micelle is bonded in a varying measure to its own solid particle. In an electric field, a part of the ions neutralizing the basic charge of the particle move with it, external, loosely bonded areas of surrounding ions move due to their opposite charge in the opposite direction. The behavior of particles in a suspension is determined essentially by the value of the so-called electrokinetic potential (zeta potential) which is electric potential of the surface of the micelle's ion shell with a fixed bond (moving with it in relation to the dispersion medium). If it is considerably higher than zero, the micelles appear in mutual collisions electrically charged and repelling forces prevent coagulation. When the zeta potential is equal to zero (i.e., the entire ion shell has a fixed bond to the particle and moves along with it), the particle behaves as neutral and coagulation occurs easily. Such a case applies to multivalent ions when relatively small ions with a strong charge can easily enclose a particle in a neutralizing shell. The opposite behavior is shown by monovalent ions (with the exception of the extraordinarily small ion H^+) which form a loosely bonded voluminous cloud of the charge in the vicinity of the particle and account for a relatively high value of the electrokinetic potential. Coagulative efficiency of ions increases with their valency. If we want to suppress coagulation of particles and

achieve liquification of the suspension we must substitute the original small multivalent ions by the larger monovalent ions.

Another way to improve the efficiency of deflocculation agents is to use protective colloids. A kaolin particle covered by a layer of adsorbed polyelectrolyte behaves as if it were made up in its entirety of that material and a system of such particles is much less sensitive to coagulation [2-7].

Material and Methods

Preparation of a steatite suspension with a high contents of dry matter while remaining at the same time fluid enough to be suitable for spray drying is a demanding task [8]. Steatite contains:

1. minimally 70% of talc substrate from which Mg^{++} ions are released by grinding,
2. dolomite from which Mg^{++} and Ca^{++} ions are extracted during grinding,
3. highly plastic ceramic materials classified as difficult to liquify,
4. talc materials belonging due to their granulometric and granulomorphic properties to substances promoting increased viscosity.

We attempted to liquify a steatite suspension by means of domestic liquifying agents (24), textile dispersing agents (18) and available deflocculants from abroad (21). The amount of the introduced dispersion agent was selected as follows:

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a) individual liquifying agents were added in amounts of 0.1% each from 0% to 1% and by 0.2% from 1% to 2%

b) where two or more liquifying agents were combined, except for one instance, all were added in constant amounts which corresponded to the previously determined on the liquification curve and the remainder was graduated by 0.1% from 0% to 1%.

Liquification tests were conducted in accordance with Czechoslovak state norm (CSN) 72 1087 [9] in a manner suitable for high concentrations: 250 g of dry ground and screened steatite were weighed and placed into a $\frac{1}{2}$ l PE bottle with a corresponding amount of distilled water, dispersion agent and 250 g of grinding balls of approximately 18 mm diameter. The mixture was thoroughly homogenized and subsequently agitated for 1.5 h in a horizontal shaker at 126 vibrations/min. After 20 hours of rest the mixture was again agitated for 1 h. Apparent viscosity was measured immediately after agitation by:

--a rotary viscosimeter (CSN 72 1087)--at one velocity stage ($D = \text{approximately } 100 \text{ s}^{-1}$),

--a flow-through cup (CSN 67 3013) with a water equivalent heat capacity of 10.2 s (100 ml).

At optimum liquification we determined: liter density/weight, dry matter, pH, temperature, foaming quality (quantitatively), sedimentation and chronological stability.

Chronological stability of a suspension denotes the stability of apparent viscosity after 1, 3 and 5 days of maturing. The suspension was agitated for 1 h prior to measurements. This test supplemented the tixotropy measure mentioned in literature [4, 9]. Chronological stability determined by the above mentioned method better expresses (from the operational viewpoint) suitability of the suspension for spray drying, because:

- a) the suspension is in motion during spray drying,
- b) the suspension need not be dried immediately after grinding.

Apparent dynamic viscosity was determined by a rotary viscosimeter only at a single speed, but always under identical conditions. Measurement of apparent viscosity was supplemented by determining the flow-through time of the suspension through the cup. Determination of these two values is not time consuming and from the attained results it is possible to determine with adequate precision the suitability of the suspension for spray drying even in less well equipped production plants.

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Liquification Tests

Apparent viscosities of nonliquified concentrated suspensions are shown in Figure 1 (curve # 1 and 1'). With a view to the requirement for preparation of suspensions with a high contents of

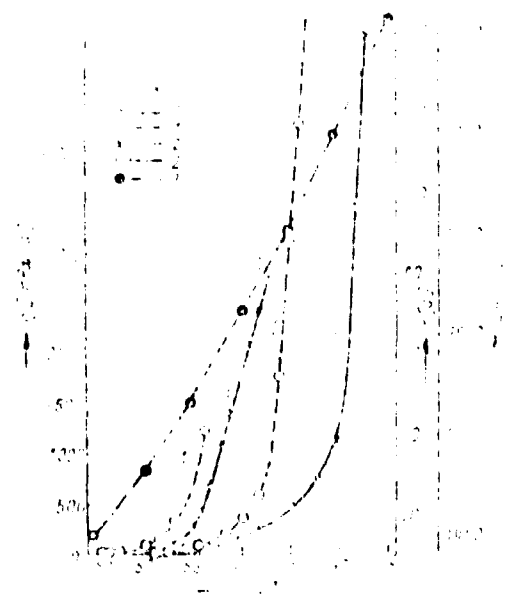


Fig. 1. Apparent dynamic viscosity of an operational steatite suspension and its liter/weight density in relation to contents of dry matter.

1 - without liquifiers, measured by a viscosimeter; 1' - without liquifiers, measured by flow-through cup; 2 - with a mixture of liquifiers, measured by a viscosimeter; 2' - with a mixture of liquifiers, measured by cup; 3 - liter weight/density of the suspension (L II) (g/l); C - dry matter contents in the suspension (%); η - apparent dynamic viscosity (mPa.s); t - flow-through time for 100 ml of suspension through the cup (s').

dry matter, we selected for the liquification tests we selected an initial concentration of 63% of dry matter for which the measured apparent viscosity was 2793 mPa.s ($D = 94.s^{-1}$) not flowing through cup.

In the subsequent phase was undertaken testing of the efficiency of individual dispersion agents described in a number of previous publications [2, 5, 10-12]. The suspension was liquified most effectively by Humitan F (sodium salt of tetrabasic humic acids) with a reading of 578 mPa.s and 99.5 s at 0.8% of liquifier. The progression of the liquification curves is shown in Figure 2 (curves # 3 and 3'). Suspensions with other dispersion agents showed either higher viscosity, or did not flow through the cup, a sign of high tixotropy. To supplement the data, Figure 2 also shows the viscosities for 60 and 55% suspensions (curves # 2 and 2') where minimum liquification curves were obtained through lower amounts of liquifiers.

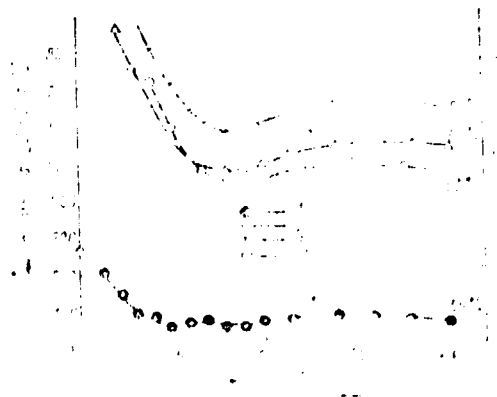


Fig. 2. Apparent dynamic viscosity of steatite suspensions with varying contents of dry matter in dependence on additive Humitan F.

1 - suspension containing 55% of dry matter, measured by viscosimeter;
2 - suspension containing 60% of dry matter, measured by viscosimeter;
3 - suspension containing 63% of dry matter, measured by viscosimeter;
3' - suspension containing 63% of dry matter, measured by cup; C - additive Humitan F (%); η - apparent dynamic viscosity (mPa.s); t - flow-through time for 100 ml of suspension through the cup (s).

Subsequently followed verification of the effectiveness of two-component liquifiers, where the first component was in all instances Humitan F in the constant amount of 0.8%. Figure 3 shows the progress of tests through which were achieved the lowest apparent viscosities /187 by:

a) hexametaphosphate of Na (curve # 3), optimum at 0.6%, 180 mPa.s and 80 s,

n) pyrophosphate of Na (curves # 4 and 4'), optimum again at 0.6%, 446 mPa.s and 70 s.

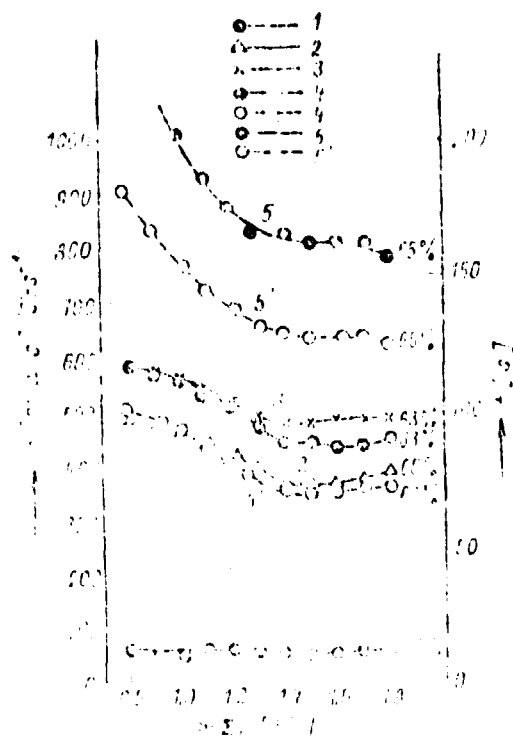


Fig. 3. Apparent dynamic viscosity of steatite suspensions with varying contents of dry matter at a constant (0.8%) addition of Humitan F in dependence on: a) addition of Na pyrophosphate (curves # 4, 4' and 5, 5'); b) addition of Na hexametaphosphate (curves # 1, 2, 3); 1 - suspension containing 55% of dry matter, measured by viscosimeter; 2 - suspension containing 60% of dry matter, measured by viscosimeter; 3 - suspension containing 63% of dry matter, measured by viscosimeter; 4 - suspension containing 63% of dry matter, measured by viscosimeter; 4' - suspension containing 63% of dry matter, measured by cup; 5 - suspension containing 65% of dry matter, measured by viscosimeter; 5' - suspension containing 65% of dry matter, measured by cup; Σ_c - sum of addition of Humitan F (0.8%) and additional liquifier (%).

The mixture with pyrophosphate proved more effective and was used for liquifying a 65% suspension--curves # 5 and 5'. Figure 3 also shows the progress of liquification of a 60% suspension (curve # 2) and 55% (curve # 4). The effects of additive liquifiers hexametaphosphate or pyrophosphate were not noticeable in the 55% suspension, probably due to an overdose of Humitan F, while the apparent viscosity of the 65% suspension kept decreasing with addition of higher amounts of dispersion agents.

Additional tests verified the effectiveness of liquifiers in a combination of 3 and 4 components, whereby the first 2 components were Humitan F (in an amount of 0.8%) and Na pyrophosphate or Na hexametaphosphate in an amount of 0.6%. The suspension used again contained 63% of dry matter. Use of 0.15% dinaphthylmetanodisulfanone Na (trade mark Kortamol NNO) and 0.25% condensation product of betanaphthosulfo acid and creosol with formaldehyde (trademark Efektan S 13) facilitated attainment of the following values of apparent viscosity:

1. mixture with hexametaphosphate Na: 371 mPa.s, cup: 57 s.
2. mixture with pyrophosphate Na: 330 mPa.s, cup: 49 s.

The progress of liquification curves resembles the curves plotted in Fig. 3.

Figure 4 shows the results of measurements of the chronological stability of suspensions with the two types of liquifiers. The progression of curves in this figure shows that apparent viscosity of the suspension containing a mixture of liquifiers with pyrophosphate kept decreasing with the length of maturing (curves # 2 and 2').



+Fig. 4. Chronological stability of steatite suspension (viscosity of the suspension in relation to its age) containing 63% of dry matter liquified by a combination of 4 liquifiers, namely: a) 0.8% Humitan F, 0.6% hexametaphosphate Na, 0.1% Kortanol NNO, 0.25% Efektan S 13 (curves # 1 and 1'), b) 0.8% Humitan F, 0.6% pyrophosphate Na, 0.15% Kortamol N2NO, 0.25% Efektan S 13 (curves 2 and 2'). 1, 2 - viscosity of the suspension measured by viscosimeter; 1', 2' - time for suspension flow-through measured by cup; S_t - viscosity of the suspension after 1, 3, 5 days; η - apparent dynamic viscosity (mPa.s); t - flow-through time for 100 ml of suspension through cup (s).

Introduction of Binders Into Granulate From Spray Drying

Granules prepared by spray drying from a suspension containing liquifiers are suitable for molding conventional types of compacts. However, they proved unsuitable for compacts calling for high strength, e.g., fuse bushings into which threading must be cut after molding. The latter products, be they made of premolded granules or from granules produced by spray drying, were reinforced by dipping into molten paraffin prior to cutting the thread. As this type of technology is unhygienic and labor intensive, we looked for a method of introducing suitable binders directly into the granules in spray drying. /188

A great multitude of various binders is currently used in technological field work. A number of published studies [13-16] attempted

to come up with a classification of binders. The preferable binders for ceramics are those with reversible (recuperative) properties (i. e., again effective in recycled blunged waste). We tested the following binders (and/or combinations of binders and plasticizers):

<u>Additive (Trademark)</u>	<u>Effective Substance</u>
Sokrat 2802	Copolymer of styrene with acrylates
Sokrat 1018	Acrylate copolymer with carboxyl groups
Sokrat 501, 508	Copolymer of vinyl acetate with acrylate
Disapol AA	Copolymer of butylmetacrylate with meta- cryn amide
Disapol M1-40	Polymethylmetacrylate (polymethylacrylane softened by dibutylphthalate)
Disapol BB 45	Copolymer of butylmetacrylate and butyl acrylate
Disapol S 10, S 20	Copolymer butylacrylate/vinylacetate softened by dibutylphthalate
Sokrat 3001, 3019, 3069	Polyacrylic acid and its ammonia salt
Tylosa S 20	Carboxymethylcellulose
Dextrin	High-molecular product of partial hydro- lysis of starch
Starch syrup	Starch polysaccharide (key components amylose and amylopectin)
Sleviol R	Polyvinyl alcohol
Spclaprof PE	Secondary polyethylene emulsion
Spolaprof PD	Aqueous paraffin emulsion
PFG 300 through 6000	Polyethylene glycols
Slovasol 2130	Ethylene oxide
Apretar RV	Polyethylenoxide wax
Resol F, LB 1, LB 2	Phenol-formaldehyde resin
Dukol A, M	Urea-formaldehyde resin
Umacel C	Urea-formaldehyde resin
Umaform A	Urea-formaldehyde resin
Depremol DT	Esterified urea-formaldehyde precondensate
Sicofan 1	Melamine-formaldehyde resin
Ligra 1	Pulp mill waste
Ligrafo ASK	Sulfone waste (lignin sulfonate).

However, none of the enumerated binders proved completely satisfactory. The individual binders showed in particular the following shortcomings:

1. failure to improve mechanical strength to the requisite level,
2. causing the suspension to foam,
3. loss of effectiveness in spray drying,
4. adequate improvement of strength, but coagulating the suspension.

For further experiments were selected binders belonging to the 4th group and we sought for additional possibilities for reducing apparent viscosity to compensate for their adverse effects.

Further reduction of the apparent viscosity of a suspension can be achieved mainly by 2 methods:

- a) increased temperature,
- b) mixing, i.e., constant interference with the structure.

Effects of temperature on the apparent viscosity of a 65% suspension are shown in Figure 5. Progression of curve 1 shows that an increase in temperature of the suspension by 40°C represents an approximately 60% decrease in viscosity. Curve # 1' in this figure represents "viscosity" (outflow time) of the suspension measured by a flow-through cup. Its progression shows that this method can be used to measure suspensions up to a temperature of 35°C. However,



Fig. 5. Apparent dynamic viscosity of a 65% steatite suspension in dependence on temperature.
1 - suspension measured by viscosimeter;
1' - suspension measured by flow-through cup;
 θ - temperature of the suspension ($^{\circ}\text{C}$);
 η - apparent dynamic viscosity (mPa.s);
t - flow-through time for 100 ml of suspension through the cup.

reduction of apparent viscosity of the suspension can be achieved under operational conditions only at the expense of additional investment costs and considerable consumption of energy. For this reason we decided to use the other method---mixing of suspensions [17]. The effects of mixing on the apparent viscosity of a 65% suspension are shown in Figure 6. It shows that if we mix the suspension for 6 hours after discharge from the drum, its viscosity decreases from about 460 mPa.s

to approximately 130 mPa.s and the time of passage through the flow-through cup becomes reduced from 105 s to approximately 30 s. Additional mixing of the suspension produces only a little change in the viscosity of the suspension.

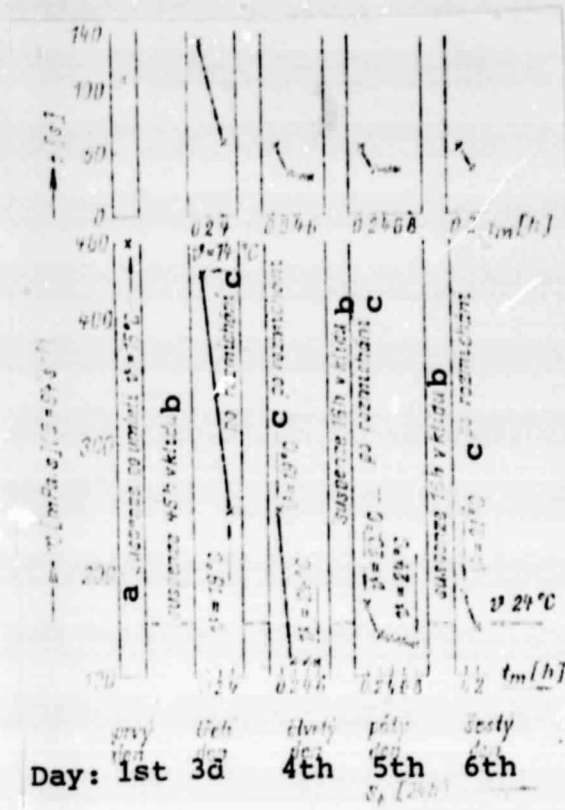


Fig. 6. Effects of mixing on the apparent viscosity of a 65% steatite suspension (illustrative representation).

t_m - time for mixing the suspension by a propeller agitator (h);

S_t - age of suspension (24 hrs);

η - apparent dynamic viscosity (mPa.s);

t - flow-through time for 100 ml of suspension through the cup;

a - suspension after grinding;

b - suspension after 24 hrs of rest;

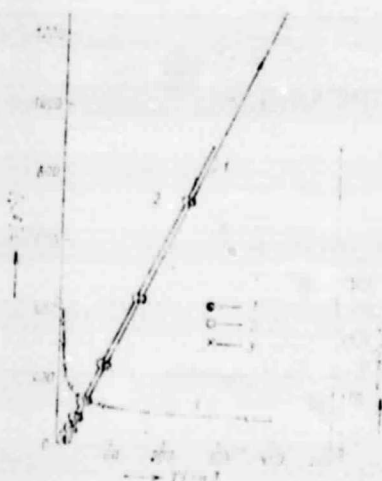
c - after mixing

We added 0.1% PVA and a 0.35% paraffin dispersion to the agitated suspension the apparent viscosity was reduced through mixing by approximately 70%. Viscosity of the suspension registered a several-fold increase, but through further mixing it again decreased to a value making it suitable for spray drying. Measurements on little frames molded from granules containing these additives showed an increase in mechanical strength in loose state by approximately 150%. The fuse bushings molded from these granules could be directly (without dipping in paraffin) subjected to cutting threadings into them.

Rheological Characteristics of Operationally Prepared Suspension and Moldability of Steatite Granulate With Chemical Additives

Rheological properties of a steatite suspension containing 67% of dry matter and chemical additives, which was operationally prepared in

mills MBM 1900, was subjected prior to spray drying to examination by the rotary viscosimeter Reotest 2, specifically in the range of the velocity gradient D from 3 s^{-1} to 1312 s^{-1} and at a temperature of 25°C . Figure 7 shows graphically the dependence of D (τ) (curves # 1 and 2) and η (τ) (curve # 3), i.e., dependence of the velocity gradient on tangential stress τ and dependence of the apparent dynamic viscosity coefficient η on tangential stress. The progression of curves D (τ) shows that the suspension has a mildly tixotropic character throughout the range of measurements; at a given velocity gradient it shows dependence of tangential stress on the duration of the effects of this magnitude [18]. Flux limit determined by graphic extrapolation: τ_0 1.2 Pa, rheological characteristic K 1.31 (η_D 20 550 mPa.s/ η_D 100 410 mPa.s), tixotropy level after 20 min of rest 0.96; pH 9.2. The liter weight/density of this suspension was 1741 g/l which shows good coincidence with its theoretically computed value - (see Fig. 1, curve # 3). In Fig. 1 are also plotted the apparent viscosities of liquified suspensions (curves 2 and 2'). A comparison of the progression of curves 1, 1 and 1', 2' in this figure shows that the suspension liquified by a mixture of liquifiers contains, at approximately identical fluidity, up to 10% more of dry matter than a nonliquified suspension. All the properties of the concentrated steatite suspension and, subsequently, also of the granulate



*Fig. 7. Rheological curves of a 67% steatite suspension measured in a rotary viscosimeter of the type Reotest 2.
1 - dependence of tangential stress (τ) on the rate of deformation (D) at increasing revolutions. 2. dependence of tangential stress (τ) on the rate of deformation (D) at decreasing revolutions. 3. dependence of apparent dynamic viscosity on tangential stress.
 τ - tangential stress (Pa).
 D - velocity gradient of deformation (s^{-1}).
 η - apparent dynamic viscosity (mPa.s).

and of the compacts were achieved only at the cost of introducing a large amount of dispersion agents (1.8 % of dry matter by weight).

The carried out tests showed that liquifiers in combination act selectively, i.e., affecting primarily that component which shows sensitivity to a ceratin type of liquifier. The results also indicate the need for a protective colloid. The amount of dispersion agents used should not, according to some literary data [19, 20], exceed 0.5%; other authors admit to potential use of higher amounts of dispersion agents [2, 21, 22].

Granules with the listed chemical additives become perfectly compacted during molding and quality of molding is also enhanced. With an improvement in the transport of material in the molding devices there also occurred improved filling of space in the molding device and forcing-out of air from spaces between granules. The attained improved plastic flow and, thus, improved compactness of compacts, was used with advantage for products into which threading must be cut after molding, so that the previous treatment of the products with paraffin prior to cutting the thread was eliminated. Reduced resistance of the molded material against flux also reduced friction against walls of the mold which produced a significant decrease in jamming, particularly of larger products, in the molding devices. Wear on the molding devices was also reduced and consumption of cutting tools in subsequent processing, e.g., turning or grinding, was also reduced. The products were uniformly molded and had a uniform volume weight. Shrinkage in firing was uniform and the products did not become deformed. Economy of molded good production was significantly improved by a higher number of molded products, reduced jamming of products in molding devices and /190 a lower rejection rate of compacts. Improved plastic flow of the molding mixture made it possible to achieve an improved dimensional precision of compacts, a fact which was used with advantage for a number of products where additional processing after firing, such as finishing by grinding, etc., was decreased or even eliminated. An "authorship certificate" [23] was awarded for the specified chemical additives as well as technological processes.

Conclusion

Tests were made of a combination of 4 liquifiers with selectively acting dispersion effects for preparation of a highly concentrated

steatite suspension with apparent viscosity suitable for spray drying. This suspension was used for preparing in a fountain drier of the type F 600, produced by the Skoda plant in Klatovy, a granulate with bulk/density weight of almost 1,000 g/l. Use of a combination of organic substances with binding and plastification properties provided the basis for molding of products capable of undergoing even the most demanding finishing operations. Actual utilization extending over 5 years already has shown the suitability of the proposed additives and technological processes. The attained results show that:

1. Suitable steatite liquifiers are Humitan F, pyrophosphate or hexametaphosphate Na, Kortamol NNO and Efektan S 13.
2. The mentioned dispersion agent mixture was used for preparation of a suspension containing 67% of dry matter and with rheological properties suitable for spray drying.
3. The tested foreign dispersion agents did not promote achieving of a lower apparent viscosity of the suspension.
4. Efektan S 13 was the only one among the tested chemicals that showed both deflecculation as well as binding effects.
5. The chronological stability of the concentrated steatite suspension (i.e., apparent viscosity of the suspension in relation to the period of maturing) with the specified chemical additives is operationally suitable.
6. The effect of mixing on reducing the apparent dynamic viscosity of the ground suspension--lowering viscosity by up to 70%--was proven in laboratory tests and used in operation.
7. Compacts made of granules prepared by spray drying show shrinkage identical to that of granules prepared by premolding, which makes it possible to use the original molding devices.
8. A mixture of PVA and paraffin dispersion introduced into the steatite suspension improved the mechanical strength of compacts in

green state by up to 250%, a fact which was used with advantage for products that must be provided with cut threadings after molding and eliminating the previously called for reinforcing of such compacts by dipping in molten paraffin.

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